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COMPARISON OF COMMON SOLVENT EVAPORATION TECHNIQUES IN ORGANIC ANALYSIS

KEY WORDS: Solvent evaporation, sample concentration organic analysis

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ABSTRACT

Isolation of organic constituents from water frequently involves an extraction with a large volume of organic solvent which must be reduced to achieve the desired sensitivity. The objective of this research was to evaluate common solvent evaporation techniques to determine which are acceptable for use in pollutant analysis. Techniques for solvent reduction from 200 to 10 mL (macro) and 8 to 0.2 mL (micro) were evaluated. The macro concentration techniques included Kuderna-Danish (K-D) concentra-

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tion, rotary evaporation, evaporation on a hot plate in an Erlenmeyer flask, and heated nitrogen blowdown in an Erlenmeyer flask. The micro techniques included micro K-D concentration, nitrogen blowdown, and nitrogen blowdown with a modified Snyder column. A series of model compounds covering a wide range of boiling points (108° to 323° C) and polarity was used. Based upon recovery efficiency alone, nitrogen blowdown was significantly superior to the other macro techniques; however, its extreme slowness gave K-D the highest Figure of Merit. With methylene chloride as the solvent, nitrogen blowdown with a column was the superior technique.

INTRODUCTION

Isolation of organic constituents from water frequently involves large quantities of solvent. Prior to instrumental analysis, the volume of solvent must often be reduced to achieve the desired sensitivity. Ideally, reduction of the solvent should be effected without loss of solute. Several methods have been used, including rotary evaporation, vacuum evaporation, free evaporation and evaporation in a Kuderna-Danish concentrator, with the latter being, by far, the most popular.

Solvent removal via rotary evaporation basically involves vacuum distillation of the solvent from a revolving flask. Polynuclear aromatics (PNA's) have been concentrated from cyclohexane by this technique.[1] Sporadic, violent boiling ("bumping") is a problem but can be controlled by employing careful conditions of rotation speed, flask geometry, temperature and vacuum. Thompson

et al.[2] effectively removed methylene chloride from pesticide extracts by slow rotation of a Kuderna-Danish (K-D) flask and receiver at a 20° angle, 125 mm Hg and 35° C. Rotary evaporation can be used only for compounds of low volatility and high stability as the evaporation of solvent from the walls of the flask may expose a large area of solute to evaporation.

Vacuum evaporative methods are similar to rotary evaporation except that much smaller solvent volumes are involved. These methods differ only in the mode of agitation and heating of the sample. The agitation serves to decrease the film thickness of the fluid, increase the surface area exposed to the vacuum, and reduce bumping during release of dissolved gases.[3] Several types of agitation have been used, including centrifugation, vortexing and shaking. Solvent removal by vacuum centrifugation generally involves 1 to 5 mL, centrifuged at 1200 or 2000 rpm under an applied vacuum. Infrared heating can be used to increase volatility. With most commercial evaporators, the samples are not visible during evaporation, so the process must be periodically interrupted to check the solvent volume. As with rotary evaporation, vacuum evaporation techniques suffer from solute losses by volatilization and are therefore applicable only to solutes of low volatility.

The Kuderna-Danish evaporative concentrator (K-D) consists of a pear-shaped flask, a small, graduated receiver and a Snyder or Vigreux column on top of the flask. The normal capacity of a K-D flask is up to 500 mL; however, micro-scale apparatus is available for concentration of small volumes (<10 mL). The

solvent is boiled using a steam bath or other heat source. The small chambers in the Snyder or Vigreux column cause multifaceted refluxing which allows only evaporation of the most volatile component, generally the solvent. Solvents with boiling points less than approximately 95° C can be evaporated using a steam bath. Higher boiling solvents (e.g., toluene) have been evaporated from a flat-bottomed boiling flask topped with a Snyder column using a hot plate;[4] even though this can constitute a safety hazard. K-D concentration has been used for many different solute-solvent systems. The most successful concentrations occur when there is a large difference in the volatility of the solute and solvent, such as Kepone® in methanol/benzene,[5] pesticides and PCB's in methylene chloride/hexane,[6] and polychloronaphthalenes in toluene.[4] As the volatility of the solute increases, so does the potential for evaporative losses. Only 10-20 percent of Aroclor 1016 was lost during evaporation of methylene chloride[7] while 58 percent of trichloroethylene was lost during evaporation of Freon TF®.[8] It has been suggested that such losses can be assessed and compensated for by inclusion of internal standards prior to evaporative concentration with the condition that the standards be similar in volatility to the compounds being determined. Junk et al.[9] have performed a rather extensive study of parameters that affect solute recovery during K-D concentration of compounds of various chemical classes from diethyl ether. The mode of heating, surface activity of glassware, use of boiling chips or type of column (Snyder or Vigreux) did not affect the solute recovery. However, vessel shape was significant since continuous rinsing of the interior walls of the flask by the solvent is critical in minimizing evaporative

solute losses. The rate of evaporation also affects solute recovery: higher losses of priority pollutants have been observed at slower evaporation rates than at faster rates.[10] An indication of the reliability of the technique is that it has been the recommended technique for several years for the analysis of wastewaters for priority pollutants.[11]

Free evaporation (nitrogen blowdown), where solvent is removed by blowing a gas stream over the liquid surface, is predominantly used for further evaporation of small solvent volumes and is applicable only to solutes of very low vapor pressure. Losses as high as 80% have been observed during this technique.[7] In a study of recovery of chloronaphthalenes during concentration of toluene solutions, losses of 68, 77, 39 and 25 percent mono-, di-, tri-, and tetrachloronaphthalenes, respectively, were observed at 45° C whereas no significant losses were observed at 24° C. Previous studies indicated that a more efficient concentration of small volumes can be achieved with a micro-scale K-D apparatus.[5]

Since the solvent evaporation is widely used to concentrate organic extract samples and the potential for solute loss is high, validation of the solvent evaporation step is imperative. The objective of this research was to evaluate several common solvent evaporation techniques.

EXPERIMENTAL

Materials

All solvents were Distilled in Glass®, obtained from Burdick and Jackson, Inc., Muskegon, MI. Chemical compounds were obtained

as follows: 2-chloroethylvinylether and fluorene, Aldrich Chemical Co., Milwaukee, WI; lindane and 1-nitrobutane, Pfaltz & Bauer, Inc., Stamford, CT; bis(2-chloroethyl) ether, Chem Service, Westchester, PA; phenol, Mallinckrodt, St. Louis, MO; and m-dichlorobenzene, EPA Pesticide Repository, HERL, Research Triangle Park, NC. The gas chromatographic column packing, 1 percent SP 2250 on 100/120 mesh Supelcoport, was obtained from Supelco, Inc., Bellefonte, PA. Standard solutions were prepared in methylene chloride such that the final concentration ranged from about 120 to 200 μg/mL following the solvent evaporation.

Kuderna-Danish Evaporation

The solution (200 mL) was placed in a 500-mL K-D flask equipped with a 10-mL receiver tube and a Snyder column prewet with 1 to 2 mL of methylene chloride. Details of the apparatus and its operation are described elsewhere.[12] The evaporation was performed on a steam bath at 75° to 85° C and completed in 10 to 15 min. The apparatus was removed from the steam bath when the volume reached approximately 5 mL and allowed to cool for at least 10 min before removing the column. The receiver tube-flask joint was rinsed with solvent into the concentrator tube and the volume brought to 10 mL. It was found that several precautions must be taken during K-D evaporation. To keep the solution from bumping, the addition of micro boiling chips is important. The chips are of 30/60 mesh marble and are extracted with methylene chloride prior to use. Insulation with open-ended tin cans, serving as chimneys, around the K-D flasks and aluminum foil around the Snyder column helps maintain a steady evaporation process. Snyder

columns from different manufacturers varied slightly in design and therefore in performance. With Lab Glass columns, the glass ball sits in a deeper indentation and is subject to a greater chance of sticking and disrupting smooth evaporation. Snyder columns from Ace glass did not exhibit these problems.

Rotary Evaporation

The rotary evaporator used was a Buchler Model PF-1029 equipped with a bump trap and a 500 mL round-bottom flask. Evaporations of 200 mL were done under water aspirator vacuum at ambient temperature with a rotation speed of about 80 rpm and were completed volume ~5 mL) in 15 to 20 min. The sample was then transferred to a graduated cylinder and the volume brought to 10 mL with rinsings from the round-bottom flask. No special precautions were required with this method.

Hot Plate Evaporation

The hot plate evaporations were performed in a 500 mL Erlenmeyer flask by gently boiling the solvent. The volume was reduced from 200 mL to approximately 5 mL in 30-35 min. The sample was transferred to a graduated cylinder and the volume brought to 10 mL with solvent rinsings of the Erlenmeyer flask. No special precautions were required with this method.

Nitrogen Blowdown

A 500-mL Erlenmeyer flask containing 200 mL of the model compound solution was heated slightly (30° C) and the volume reduced to approximately 5 mL in 120 min under a gentle stream (about 350 mL/min) of dry nitrogen. The sample was then transferred to a graduated cylinder and the volume brought to 10 mL

with rinsings of the Erlenmeyer flask. No special precautions were required with this method.

Micro Kuderna-Danish

An aliquot of the standard solution was added to 8 mL of solvent in a 10-mL K-D concentrator tube, previously calibrated at 0.20 mL because the manufacturer's marks were found to be inaccurate (9 percent relative standard deviation). Three 35/60 mesh marble boiling chips were added. A four ball micro Snyder column was used. Ten to twenty min were required to evaporate the solvent on a steam bath. The apparatus was held by hand, since frequent agitation was required to prevent bumping of the sample. Evaporation was terminated when the concentrator tube contained less than 0.1 mL. After cooling for 10 min, the column was removed and the volume adjusted to 0.20 mL. Samples were transferred to a 2-mL vial, sealed with a Teflon®-lined screw cap, and stored at 4° C.

Nitrogen Blowdown With Column

An aliquot of the standard solution was added to 8 mL of solvent in a 10-mL K-D concentrator tube. A modified (no balls) Snyder column was used. The concentrator tube was placed in a 30° C water bath while the volume was reduced from 8 mL to 0.20 mL under a gentle stream (about 350 mL/min) of dry nitrogen directed on the surface of the solvent with a Pasteur pipet. The time required was 10 to 15 min. The sample was transferred to a 2-mL vial and stored at 4° C.

Nitrogen Blowdown Without Column

The procedure was the same as nitrogen blowdown with column except no column was used.

Analysis

The concentrates were analyzed on a 180-cm by 2-mm i.d. glass column packed with 1 percent SP 2250 on 100/120 mesh Supelcoport. A Varian 3700 GC with on-column injection and a flame ionization detector was used. The injector and detector temperatures were 150° C and 300° C, respectively. The column oven was maintained at 35° C for 2 min and then programmed to 200° C at 8° C/min. Hydrogen flow was 30 mL/min, air flow was 300 mL/min, and the nitrogen carrier flow was 30 mL/min. Injection volume was 1.0 μL. Manual integration (peak height × width at half-height) was used to determine the peak area.

RESULTS AND DISCUSSION

Macro Techniques

The recovery data from the macro techniques are summarized in Table 1. The significance of the data is tabulated in Table 2. Surprisingly, nitrogen blowdown is significantly superior to other techniques in both instances.

Micro Techniques

The recovery data for the micro evaporation techniques are summarized in Table 3. To verify that the results obtained were reproducible, two sets of 5 samples each (15 percent methylene chloride/hexane) were evaporated on two separate days using nitrogen blowdown with column as presented in Table 3. The significance of the difference in recoveries between the two data sets was only at 70 percent (Table 4). This suggests a high degree of reproducibility.

TABLE 1
Summary of Recoveries Using Macro Evaporation Techniques[a]

Compounds	Recoveries (%) Kuderna Danish	Rotary Evaporation	Hot Plate Evaporation	Nitrogen Blowdown
Methylene Chloride				
2-Chloroethylvinylether	ND[b,c]	ND	72(12)	ND
1-Nitrobutane	ND	ND	78(17)	ND
m-Dichlorobenzene	85(7)[d]	78(4)	77(10)	83(6)
Bis(2-chloroethyl)ether	74(14)	58(9)	74(12)	76(9)
Phenol	89(11)	83(5)	82(21)	101(14)
Fluorene	85(9)	75(15)	82(16)	92(10)
Lindane	84(13)	70(24)	104(22)	101(10)
Mean	83(7)	73(14)	81(14)	91(12)
Time Required (min)	10-15	15-20	30-35	120
15% Methylene in Hexane				
2-Chloroethylvinylether	ND	ND	ND	ND
1-Nitrobutane	84(14)	61(17)	ND	ND
m-Dichlorobenzene	83(4)	80(8)	83(5)	91(7)
Bis(2-chloroethyl)ether	67(5)	70(13)	70(16)	86(8)
Phenol	59(14)	68(22)	54(22)	62(5)
Fluorene	92(4)	100(4)	96(5)	95(3)
Lindane	90(2)	111(6)	98(3)	98(6)
Mean	76(16)	82(24)	80(23)	86(16)
Time Required (min)	10-15	15-20	30-35	120

[a]Mean recovery (%) of five replicates.

[b]No data.

[c]2-Chloroethylvinylether peak lost in solvent peak.

[d]Relative standard deviation.

TABLE 2
Significance of Differences in Recoveries
Between Macro Evaporation Techniques[a]

Techniques	Methylene Chloride	15% Methylene Chloride/Hexane
Nitrogen/Kuderna Danish	90	58
Nitrogen/Hot Plate	83	92
Nitrogen/Rotary	97	82
Kuderna Danish/Hot Plate	<10	96
Kuderna Danish/Rotary	>99	29
Hot Plate/Rotary	71	17

[a]Probability that difference in values is real. Significance tested using a paired "t" test where the mean recoveries for each compound are paired.

TABLE 3
Summary of Recoveries Using Micro Evaporation Techniques[a]

Compound	Recoveries (%)			
	Micro Kuderna-Danish	Nitrogen Blowdown with Column		Nitrogen Blowdown without Column
Methylene Chloride				
2-Chloroethylvinylether	ND[b]	ND		ND
1-Nitrobutane	47(8)	57(9)		56(4)
m-Dichlorobenzene	76(5)	88(8)		77(10)
Bis(2-chloroethyl)ether	77(3)	87(8)		78(5)
Phenol	77(5)	89(9)		81(4)
Fluorene	91(8)	89(4)		81(4)
Lindane	74(11)	96(4)		85(6)
Mean	74(19)	84(17)		76(13)
Time required (min) each	10-12	10-15		10-15
15% Methylene Chloride in Hexane				
		Set 1	Set 2	
2-Chloroethylvinylether	ND	-	-	ND
1-Nitrobutane	ND	-	-	ND
m-Dichlorobenzene	61(3)	24(8)	26(8)	30(23)
Bis(2-chloroethyl)ether	93(3)	77(5)	70(19)	77(3)
Phenol	81(14)	36(6)	36(25)	50(12)
Fluorene	90(7)	85(5)	80(7)	86(5)
Lindane	78(9)	92(4)	92(10)	94(7)
Mean	81(16)	63(49)	61(41)	67(40)
Time required (min) each	25-30	10-15	10-15	10-15

[a]Mean recovery (%) of five replicates, number in parentheses is relative standard deviation.

[b]No data, 2-chloroethylvinylether and 1-nitrobutane peaks lost in solvent peak.

TABLE 4
Significance of Differences in Recoveries
Between Micro Evaporation Techniques[a]

Techniques	Methylene Chloride	15% Methylene Chloride/Hexane
Micro K-D/Nitrogen with Column	>99	89
Micro K-D/Nitrogen without Column	>99	82
Nitrogen with Column/Nitrogen without Column	50	97
Set 1/Set 2	-	68

aProbability that difference in values is real. Significance tested using paired "t" test where the mean recoveries for each compound are paired.

Problems were encountered in micro K-D concentrations using 15 percent methylene chloride/hexane. The solvent volume could not be reduced to the desired 0.2-mL volume. At a volume of 1.0 to 0.5 mL, the column flooded and the concentrator tube became dry. As the column cooled, the solvent drained back into the concentrator tube. Aluminum foil wrapped around the column had no noticeable effect. Even after 25 to 30 min of evaporation, the level could not be reduced beyond 0.5 to 1.0 mL. The final volume achieved was used for determining recoveries.

A test of the significance of differences in recoveries between the three techniques with methylene chloride (Table 4) indicated that nitrogen blowdown with a column is superior to the other two techniques. On the other hand, with 15 percent methylene chloride/hexane, the results are much less clear (Table 4). It appears that micro K-D gives recoveries superior to the other

TABLE 5. Figure of Merit Estimations for Solvent Evaporation Techniques

Technique	Recoveries (%)[a] MC	Recoveries (%)[a] MC/Hex	Time (min)	Number Simultaneously	Time Sample (min)[b]	Ease of Operation[c]	Cost/Unit[d]	Figure of Merit[e] MC	Figure of Merit[e] MC/Hex
MACRO									
Kuderna-Danish	84	79	10-15	3-4	3.6	5,4	110	74.8	69.8
Rotary Evaporation	74	82	15-20	1	17.5	3,4	607	46.9	54.9
Hot Plate	81	80	30-35	6-8	4.8	4	57	71.6	70.6
Nitrogen	91	86	120	6-8	17.5	5	121	67.3	62.3
MICRO									
Micro K-D	74	81	10-12 (25-30)[f]	1[g]	11 (27.5)[f]	5,3 (9)[f]	85	58.2	43.7
Nitrogen/Column	84	62	10-15	6-8	1.85	2	94	79.2	57.2
Nitrogen	76	67	10-15	6-8	1.85	2,2	74	71.4	62.4

[a] MC = methylene chloride; MC/Hex = 15 percent methylene chloride/hexane.

[b] Average time/average number simultaneously.

[c] Subjective evaluations of experienced analysts, with 1 = easiest and 10 = hardest.

[d] Total cost (U.S. dollars) of glassware and apparatus based on recent catalog prices. Where appropriate, cost of multiple units (such as large steam baths) is divided into a unit cost.

[e] Figure of Merit = (% Recovery) - (Time/Sample) - (Average Ease of Operation) - 0.01 (Cost/Unit).

[f] Value in parentheses is for 15 percent methylene chloride/hexane.

[g] Only one-at-a-time, because the apparatus must be manually shaken to maintain even evaporation.

techniques, although not to the degree observed for nitrogen blowdown with column vs. the other techniques with methylene chloride. However, the long time and inability to achieve a 0.2-mL final volume make micro K-D unacceptable.

Figure of Merit

Table 5 presents Figure of Merit data for the solvent evaporation techniques. This table summarizes recovery data, efficiency, and cost to facilitate selection of techniques. A Figure of Merit was calculated as shown. The factor weightings are subjectively chosen. The recovery efficiency is of prime importance and was highly weighted, while apparatus cost is a one-time expense and may be amortized over the life of the equipment, roughly estimated at 100 uses.

CONCLUSIONS

It should be noted that the model compounds selected represent both those known to be effectively recovered from solvent evaporations (e.g., lindane) and those which are generally considered too volatile for this technique (e.g., 2-chloroethylvinylether and m-dichlorobenzene). Thus, many of these compounds pushed the techniques to and beyond their limits, yielding either poor or unmeasurable recoveries.

The macro evaporation techniques (200 to 10 mL) K-D, rotary evaporation, hot plate evaporation and nitrogen blowdown were evaluated. Mean recoveries of seven model compounds from methylene chloride ranged from 74 to 97 percent with nitrogen blowdown giving the highest recoveries. With 15 percent methylene chloride/hexane as the solvent, recoveries ranged from 79 to 86

percent with nitrogen blowdown having the highest and K-D the lowest recoveries. The time required ranged from 10 min (K-D) to 120 min (nitrogen blowdown). Based upon recovery efficiency, nitrogen blowdown was significantly superior to the other techniques. However, its extreme slowness reduced its Figure of Merit, making K-D the superior technique. Regardless of macro technique used, it has been found that the recoveries improve markedly with experience of the analyst. Therefore, it is recommended that neophytes be trained with several practice sets of spiked solvent before working with environmental samples. In addition, the recovery data indicate that this is the critical step with respect to analyte losses for most analyses. Therefore, rigorous quality control procedures must be followed.

The micro evaporation techniques (8 to 0.2 mL) micro K-D, nitrogen blowdown with a modified Snyder column, and nitrogen blowdown without any column were evaluated. Mean recoveries of six model compounds in methylene chloride were 74 percent for micro K-D, 84 percent for nitrogen blowdown with column, and 76 percent for nitrogen blowdown without column. The nitrogen blowdown with column was significantly superior (97 to 99 percent confidence) to the other two techniques. Mean recoveries in 15 percent methylene chloride/hexane were 81 percent for micro K-D, 62 percent for nitrogen blowdown with column, and 67 percent for nitrogen blowdown without column. The significance of the differences in recoveries was contradictory due to the variability of the data. Micro K-D had superior recovery but could not concentrate samples to the desired 0.2 mL and, therefore, could not be

given an unqualified recommendation. The comments above concerning analyst experience and quality control for macro techniques are more critical for micro techniques. The chances for loss by bumping with micro K-D and by evaporation to dryness with all techniques are much greater with micro techniques. Procedures must be carefully validated prior to use and constantly monitored during their use.

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